Canadian Mineralogist
Vol. 14, pp. 307-313 (1976)

ELECTRON MICROSCOPIC STUDIES OF SERPENTINITES

B. A. CRESSEY AND J. ZUSSMAN
Department of Geology, University of Manchester

ABSTRACT

Electron microscopy of ion-thinned samples of serpentinites has revealed a new structure made up of lizardite laths stacked in sectors to form polygonal prisms either on their own or surrounding a core of cylindrical chrysotile. The polygonal serpentine is identified with the so-called "Povlen-type chrysotile" described in the literature. Both polygonal and cylindrical fibres have unusually large diameters.

SOMMAIRE

Une étude par microscopie électronique d'échantillons de serpentinites amincis par faisceau ionique a mis en évidence une nouvelle structure qui consiste en empilements de plaques de lizardite, soit en secteurs dans des prismes polygonaux, soit comme gaine autour d'un noyau de chrysotile cylindrique. La serpentine polygonale s'identifie avec la chrysotile de type Povlen, déjà décrite. Les fibres polygonales et cylindriques sont d'un diamètre relativement large.

(Traduit par le journal)

INTRODUCTION

There have been many electron-optical studies of serpentine minerals, but so far they have not been examined *in situ* in their host rocks. Specimens in most previous work have been prepared by crushing and dispersion, destroying the original structures and orientations; the crushed grains also often adopt preferred orientations on the electron microscope grid, and not all aspects of grains are accessible to the investigator. Replicas have been used to study textures of serpentines, but cannot supply electron diffraction information. Yada (1967, 1971) studied chrysotile asbestos by embedding bundles of fibres in resin and sectioning them by ultramicrotomy. While this method may not disturb fibres drastically, it does not allow the material to be examined within its host rock. As yet this technique has not been applied to non-asbestos chrysotile.

Ion-thinning (Barber 1970; Champness & Lorimer 1971) is being used in this laboratory to prepare samples for electron microscopic studies of a variety of rocks and minerals. It permits the study of mineral grains in the orientations and with the spatial relationships in which they were formed in the rock. Since the ion-thinned specimen is taken from a normal thin section prepared as for optical microscopy, a direct correlation can be made between the electron-optical and light-optical information.

The general aim of our research project— the correlation of electron-optical, X-ray and light-optical observations on serpentinites— has not yet been accomplished. The purpose of this paper is to report unusual textural features of serpentinites, in particular a polygonal morphology which has not been observed previously, but which has been predicted (Middleton & Whittaker 1976).

DESCRIPTION OF SPECIMENS

Specimens were kindly provided from the Department of Geology and Mineralogy, University of Oxford. They have been previously studied by optical microscopy and by microbeam X-ray techniques by Wicks (1969), Wicks & Zussman (1975) and Wicks & Whittaker (in preparation), and the brief descriptions given below are based on their observations.

It should be emphasized that the mineralogy and textures of most of the specimens are complex and some of the specimens are inhomogeneous. Thus our work refers to chrysotile in some specimens where it was previously reported as only a minor constituent and in some where it does not feature at all in the sample description. (For further discussion see below).

The notations used here for the serpentine polymorphs are those used by Wicks & Whittaker (1975).

Serpentinites with pseudomorphic textures

18505, a serpentinized dunite from Setting Lake, Manitoba, Canada, consists mainly of lizardite $1T$ in α-serpentine mesh texture. Mesh rims are lizardite $1T$ and most mesh centres consist of brugnatellite with traces of lizardite $1T$; however, the mesh cells studied here consist of nearly isotropic serpentine, identified as lizardite $1T$.

18508, a serpentinized dunite from Glen Urquart, Scotland, consists mainly of lizardite $1T$ in α-serpentine mesh texture, with some brucite and lizardite in the mesh rims.

18527, a serpentinized harzburgite from Totalp, Davos, Switzerland, consists mainly of lizardite $1T$ in α-serpentine mesh texture, in which the rims are lizardite $1T$ and the mesh centres are lizardite $1T$ with lesser amounts of magnetite.

Samples examined by us from each of the above three specimens contained both mesh rims and mesh centres.

18540, a serpentinized harzburgite from Jeffrey Mine, Quebec, Canada, consists mainly of lizardite $1T$ in an α-serpentine hourglass texture.

Serpentinites with pseudomorphic textures which have begun to transform to non-pseudomorphic textures

18500, a serpentinized pyroxenite from Pipe Lake mine, Manitoba, Canada, consists mainly of lizardite $1T$ with minor chrysotile $2M_{c1}$ bastites probably after clinopyroxene, and lesser amounts of lizardite $1T$ and chrysotile $2M_{c1}$ in an α-serpentine hourglass texture after olivine.

In each of the above five specimens there were minor areas containing bastites and/or small veins of chrysotile $2M_{c1}$, but these areas were not examined in this study.

18501, a serpentinized dunite from Pipe Lake mine, Manitoba, Canada, consists mainly of chrysotile $2M_{c1}$ with lizardite in γ-serpentine mesh texture, with lesser amounts of magnetite and sulfides. Mesh rims are composed of chrysotile $2M_{c1}$ with or without traces of lizardite $1T$, and centres are composed of intimate mixtures of chrysotile $2M_{c1}$ and lizardite $1T$. Both mesh centres and mesh rims were examined in this study.

Serpentinites with non-pseudomorphic textures

18541, a serpentinized harzburgite from Jeffrey mine, Quebec, Canada, consists of two texturally distinct areas. The area examined in this study is composed of a chrysotile $2M_{c1}$ (with traces of parachrysotile) interpenetrating texture with brucite and magnetite. The specimen also contains a lizardite $1T$ hourglass texture with small amounts of lizardite $1T$ bastite and chrysotile $2M_{c1}$ (with traces of chrysotile $2Or_{c1}$ and parachrysotile) in fine veins.

18543, a serpentinized harzburgite from Jeffrey mine, Quebec, Canada, consists of two texturally distinct areas. The area examined in this study is composed of partly serpentinized (mainly lizardite $1T$) amphibole, biotite and chlorite associated with fine-grained serpentine (mainly lizardite $1T$ with traces of antigorite and chrysotile $2M_{c1}$) textures. The specimen also contains an area of very fine-grained chrysotile $2M_{c1}$ and lizardite $1T$ in γ-serpentine interlocking textures, with lizardite bastites and chrysotile veins.

Serpentine veins

18515, a serpentinized amphibole peridotite from the Lizard, Cornwall, England, contains a vein consisting of an intimate mixture of chrysotile $2M_{c1}$ and lizardite $1T$ and possibly traces of lizardite $2H$ or chrysotile $2Or_{c1}$. It appears non-fibrous in hand specimen. Generally lizardite $1T$ is more common towards the vein margins, where it is often intimately associated with brucite, and chrysotile $2M_{c1}$ is more common towards the centres. The vein (and also some slip veins of lizardite $1T$) is contained in a serpentinite consisting of lizardite mesh textures and bastites.

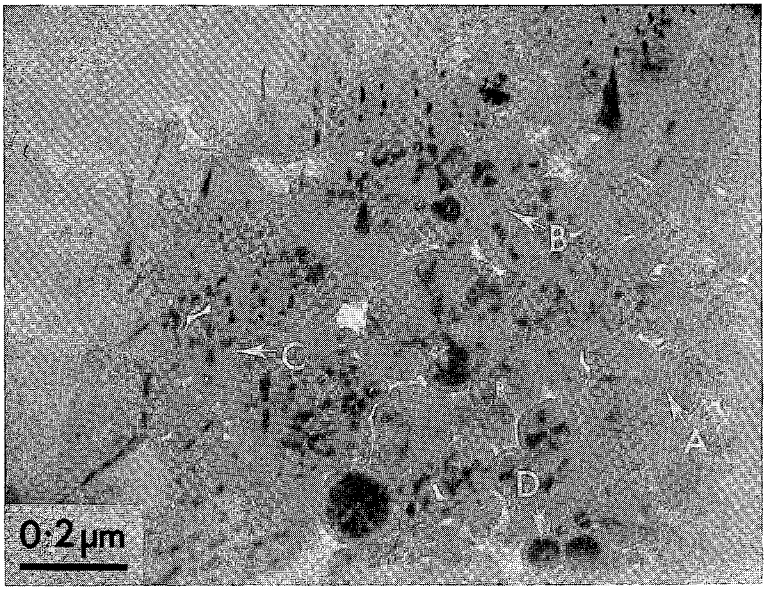
18536, a serpentinized harzburgite from Jeffrey mine, Quebec, Canada, contains a slip vein of chrysotile $2M_{c1}$ (in which Povlen-type chrysotile has been identified), passing into slip-fibre chrysotile $2M_{c1}$ with traces of lizardite $1T$ across the length of the slip face.

ELECTRON MICROSCOPIC OBSERVATIONS

18501

Specimen 18501 consists of cylindrical fibres of chrysotile in various orientations. The majority are circular in cross-section and many have diameters of about 0.06 μm but diameters range from 0.02 to 0.1 μm. They show evidence of interference during growth but they are generally complete circles (see for example A, Fig. 1). Internal diameters (i.e. the widths of hollow cylinders) are difficult to measure accurately but are mainly about 0.005-0.01 μm. The fibres appear to show image and diffraction features consistent with those published previously, but fibres with large diameters are seen to be common whereas they have been only rarely reported elsewhere (Yada 1967). The significance of chrysotile with large radii is discussed in a later section.

Near their margins almost all fibres with circular cross-sections appear to have a pale band approximately 0.005 μm wide which could be distinguished from a Fresnel fringe as it remained stationary when the image was defocused. Possibly the band contains amorphous material, but this is difficult to prove by electron-optical methods. Large fibres also show concentric, fine, pale banding which may be imperfections acquired during growth. Fibres also show radial extinction contours which are roughly parallel-sided. The contours vary in number from one pair at approximately 180° (B, Fig. 1), to two pairs at approximately 90° (C, Fig. 1), or more. Where there are many pairs, the fibre section may appear almost en-



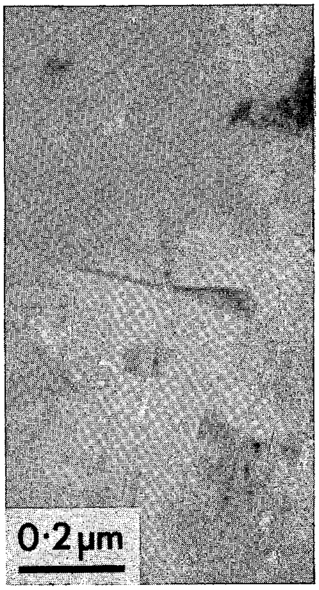


FIG. 1 (left). Various fibres seen in cross-section, showing completely circular sections (A), one pair of radial extinction contours (B), two pairs of radial extinction contours (C) and many pairs of radial extinction contours (D). Specimen No. 18501.

FIG. 2. Cross-section of a complete polygonal fibre. Specimen No. 18541.

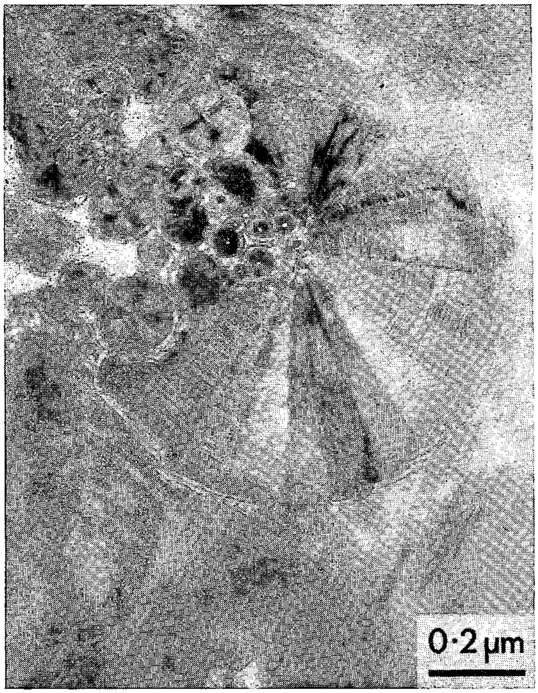


FIG. 3 (left). Cross-section of an incomplete polygonal fibre together with some normal cylindrical fibres. Specimen No. 18501.

FIG. 4. Longitudinal section of a polygonal fibre, showing core (E) and margins (F). Specimen No. 18515.

tirely black (D, Fig. 1), but still shows the pale marginal band. Such a fibre has a zone of high symmetry exactly parallel to the beam, whereas in those fibres showing only one pair of contours the zone is at a slight angle to the beam. A fibre can be seen to change from appearing entirely black, through a decreasing number of bands, to finally losing the single pair when it is tilted by about 5°.

Some fibres appear to be polygonal in cross-section and are composed of sectors (Fig. 2). The fibres are often incomplete, and their boundaries are sometimes indistinct (Fig. 3). The centres of some fibres contain normal cylindrical chrysotile about 0.05 µm in diameter. A fine concentric banding in the sectors suggests that the fibres are composed of a polygonal arrangement of flat plates, around a cylindrical core (if it is present). The radii of the polygonal fibres are variable, ranging from approximately 0.1 µm to 0.84 µm. The radii of the sectors are often variable within one fibre and usually several sectors of similar length occur close together. The number of sectors in a fibre often varies and consequently the angles between the sectors also vary. Longitudinal sections of polygonal fibres do not have the smooth outline of normal chrysotile asbestos, but are splintery with variable width along their length. Often the cores are clearly visible in these sections (Fig. 4).

Other samples

Specimens 18505, 18515, 18527 and 18541 were found to contain polygonal fibres, which were often complete, in areas of randomly-orientated chrysotile fibres. Specimens 18500, 18508, 18536 and 18540 contain incomplete polygonal fibres which often have only one or a few sectors, and they generally occur in small areas of poorly crystalline, randomly-oriented chrysotile. Only in 18543 were no polygonal fibres seen.

Although polygonal fibres often do not have cylindrical cores, they always occur closely associated with chrysotile, and are not found in areas containing lizardite but no chrysotile. The reason the chrysotile in some of the specimens was not detected by the X-ray microbeam camera is probably that in such specimens (e.g. 18540), the chrysotile only occurs in very small areas and is probably too poorly crystalline and fine-grained to diffract X-rays coherently. In support of this suggestion it has been observed that some textures, for example mesh centres after olivine, often produce fairly diffuse X-ray diffraction rings (Wicks & Zussman 1975, Fig. 6j).

CORRELATION OF ELECTRON-OPTICAL, X-RAY MICROBEAM AND LIGHT-OPTICAL OBSERVATIONS

Under the optical microscope, specimen 18501 can be seen to consist of approximately square or rectangular mesh cells which are outlined by a mesh rim of cross-fibre serpentine. Mesh centres appear to consist of mixtures of length-slow and length-fast fibres in a variety of orientations. Usually the centres are of lower birefringence than the margins and often appear isotropic. X-ray microbeam camera studies on this specimen by Wicks & Zussman (1975) indicated that rims consisted of parallel chrysotile $2M_{c1}$ fibres with occasional traces of lizardite $1T$ while the centres contain intimate mixtures of chrysotile $2M_{c1}$ and lizardite $1T$ in random orientation. Under the electron microscope these observations were confirmed and the chrysotile fibres in the rim were found to have normal dimensions. Mesh centres were found to contain chrysotile $2M_{c1}$ and polygonal serpentine fibres in apparently random arrangement. Normal (roughly equidimensional) lizardite plates were not seen. The only crystals shown by electron diffraction to be similar to lizardite were those forming the polygonal fibres, but the mode of layer stacking was not $1T$ (see next section and discussion of nomenclature).

Preliminary electron-optical observations on other samples seem to be in good agreement with the results of X-ray microbeam studies on serpentine textures by Wicks (1969), Wicks & Zussman (1975) and Wicks & Whittaker (in prep.), as summarized in the specimen descriptions.

ELECTRON DIFFRACTION STUDIES OF POLYGONAL SERPENTINE

It was not possible to obtain an electron diffraction pattern from a single sector of a polygonal cross-section, but in Figure 5, for example, patterns from a few adjacent sectors are clearly discernible. They show that the cross-sections lie approximately in the *b-c* plane (i.e. the fibre axis is approximately parallel to *a*) and that the radial direction is the *c* axis. Thus the appearance and electron diffraction patterns confirm that each sector consists of a stack of flat serpentine layers.

Diffraction patterns were also obtained from longitudinal sections of polygonal fibres. In cases where the beam was approximately perpendicular to the serpentine layers (along a fibre radius) and the section of the fibre only included one sector, the expected *hk*0 diffraction pattern was obtained (Fig. 6). It was similar to the pattern commonly obtained from a plate of lizardite lying perpendicular to the electron beam.

For those fibres in which the polygonal structure surrounds a circular core, the longitudinal sections would be expected to give an *hk*0 pattern together with *h*0*l* and *hkl* patterns superimposed upon the diffraction pattern typical of chrysotile. Such superimposed patterns were observed, sometimes associated with core and outer sections which were clearly visible in the electron micrograph. The patterns appeared to be very similar to those of Povlen-type chrysotile $2M_{cl}$; they resembled those of normal chrysotile $2M_{cl}$ but extra reflections found in the first layer line could only be indexed as 13*l*, and imply some degree of ordering in the *b* direction. In some diffraction patterns, tails of 110 reflections (and other *h*10 reflections to a less noticeable extent) had a more uneven profile than is usual for chrysotile. The subsidiary maxima were irregularly spaced, (and therefore could not be 11*l* reflections), and are thought to suggest that the fibres have an imperfect rotational symmetry in the cylindrical core.

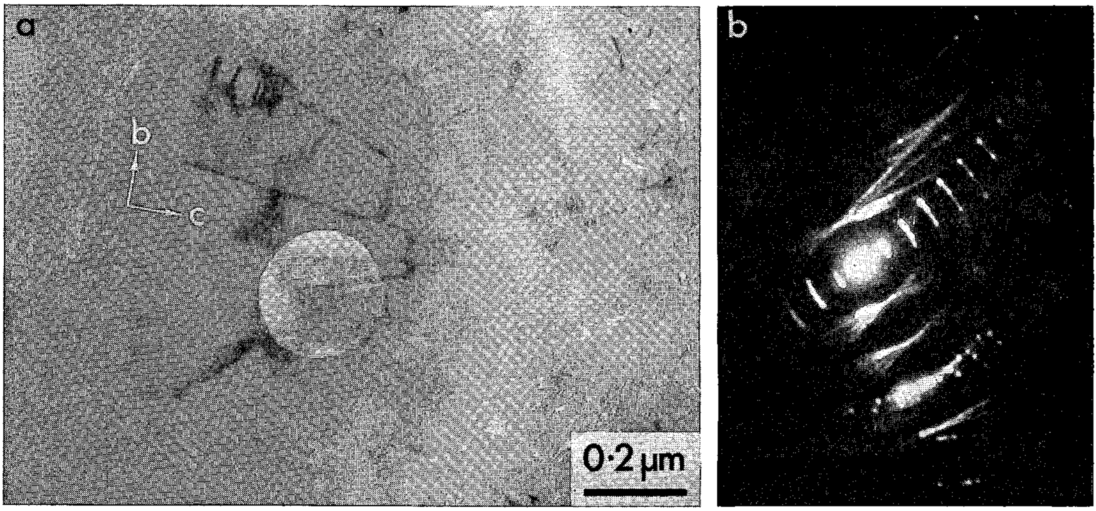


FIG. 5. (a) Cross-section of an incomplete polygonal fibre, showing the position of the diffraction aperture. Specimen No. 18501. (b) Selected-area diffraction pattern produced from the area in (a).

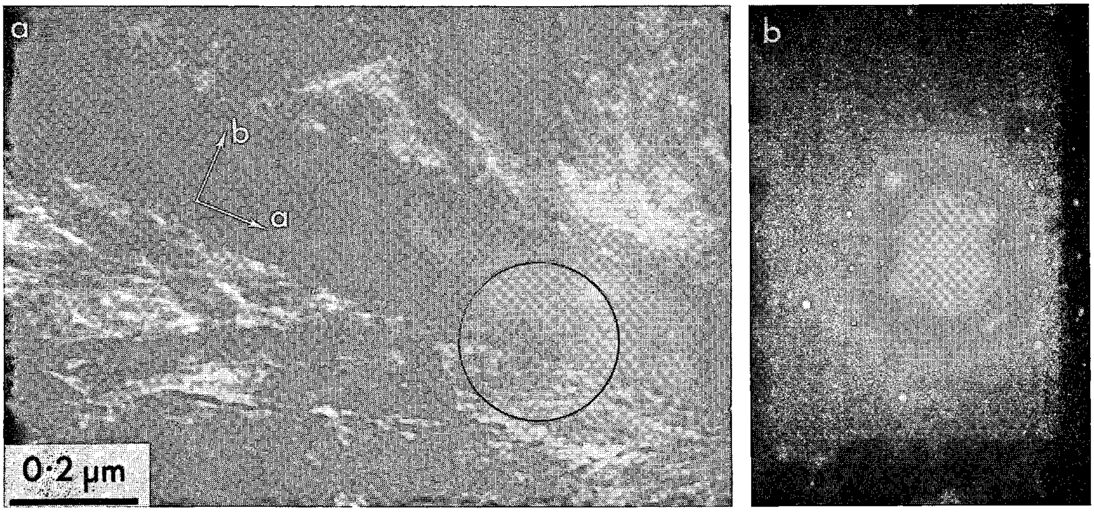


FIG. 6. (a) A single lizardite lath from a longitudinal section of a polygonal fibre, showing the position of the diffraction aperture. Specimen No. 18501. (b) Selected-area diffraction pattern produced from the area in (a).

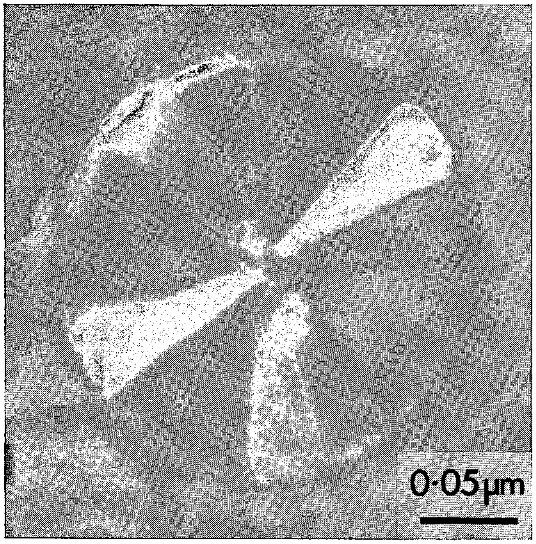


FIG. 7. Cross-section of a Povlen-type fibre. (Compare with Fig. 2). Specimen No. 0.9652.

DISCUSSION

"Povlen-type" chrysotile

The splintery chrysotile referred to as "Povlen-type" has been studied and described by Krstanović & Pavlović (1964). It has a characteristic lath-like, rather than tubular, morphology, and its electron diffraction pattern is similar to that of chrysotile $2M_{c1}$*, but it has additional reflections from *(hkl)* planes, indicating that the structure has a greater degree of three-dimensional order than the $2M_{c1}$ polymorph. Enigmatically, what appears to be a single fibril, but does not have a cylindrical lattice, nevertheless gives a diffraction pattern with approximately rotational symmetry. Middleton (1974) and Middleton & Whittaker (1976) considered several explanations for this and postulated as the most likely the existence of fibres with polygonal cross-sections either surrounded by or surrounding a core of tubular chrysotile.

We have found the polygonal structures postulated by Middleton; they sometimes occur alone and sometimes with a core of cylindrical chrysotile.

Splintery, lath-like chrysotile has already been described from electron-optical studies by Zussman *et al.* (1957), who drew attention to the presence of *hkl* reflections and rotational symmetry. (Such specimens have been described by different varietal names, e.g. schweizerite). It seemed very likely to the present investigators that this material is also similar to the "Povlen chrysotile" and to polygonal serpentine. The crushing employed by Zussman *et al.* to prepare the sample probably destroyed the polygonal structure and yielded only laths with their lengths lying perpendicular to the electron beam.

We have examined one "schweizerite" specimen (0.9652, Oxford University Museum) studied by Zussman *et al.* (1957) and subsequently described as "Povlen-type" by Middleton (1974). It contains fibres in many orientations, and those seen in cross-section are complete polygonal fibres (Fig. 7) very similar in appearance to that shown in Figure 2. Longitudinal sections give diffraction patterns similar to those previously published for "schweizerite" and "Povlen-type" chrysotile. Sample 18536, in which we found polygonal serpentine, has also been previously reported to contain "Povlen-type" chrysotile (Wicks 1969).

Nomenclature

The problem of the nomenclature of serpentine polymorphs is complicated further by the existence of polygonal serpentine. Since the polygonal prisms commonly surround cylindrical cores, perhaps they should be called polygonal chrysotile. For those described here the layer stacking is similar to that of clinochrysotile ($2M_{c1}$). We then have the anomaly of a flat-layered chrysotile. Should this material still be called polygonal chrysotile even if there is no cylindrical core? Can it be called polygonal lizardite (because it is flat-layered) even though it does not have one of the modes of stacking expected for flat layers?

Middleton & Whittaker (1976) reported a Povlen-type serpentine which does have one of the flat-layer modes of stacking (ortho-type). This could be called polygonal lizardite, without inconsistency. If this or similar material surrounded a cylindrical core the result would be a composite fibre, part chrysotile and part lizardite.

While we have not attempted an exhaustive survey of serpentinites to discover the frequency of occurrence of polygonal serpentine, our limited study has shown that it is not uncommon, and we suggest that until the above difficulties can be resolved, future descriptions refer to it as polygonal serpentine.

Chrysotile with large fibre diameters

The observation of chrysotile fibres with a greater range of diameters than has usually been reported, particularly the large ones, is

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*An orthorhombic Povlen-type chrysotile, $2Or_{c1}$, has also been observed (Middleton 1974; Middleton & Whittaker 1976).

perhaps associated with the specimen preparation technique. Crushing prior to dispersion would tend to break off the outer layers of such large fibres, leaving them to be observed as slightly curved laths. This suggestion is supported by the fact that Yada (1967), using a specimen preparation method which probably does not disturb fibres much, has observed diameters of up to 0.1 µm. However, he has stated (Yada 1971) that the outer diameter which he most frequently observed was 0.05 µm. This figure is close to the value obtained by Whittaker (1957) partly from theoretical considerations and partly from X-ray diffraction measurements. It may be significant that all the material Yada studied was chrysotile asbestos, whereas no asbestos samples were examined in this study.

Wicks & Whittaker (1975) concluded that chrysotile (curved layers) and lizardite (flat layers) have overlapping rather than distinct chemical compositions, and are therefore best regarded as polymorphs. If highly curved and flat-layered varieties are possible, then it would be surprising if layers with intermediate curvature such as those observed in the outer layers of large-diameter fibres did not exist.

Lizardite-chrysotile mixtures

Many earlier studies by X-ray diffraction and using an X-ray microbeam camera (Whittaker & Zussman 1956; Wicks & Zussman 1975) have reported serpentine specimens which are mixtures of chrysotile and lizardite. It now seems likely that in some of these the mixture could consist of cores of chrysotile surrounded by prisms of polygonal serpentine.

CONCLUSIONS

Polygonal serpentine has been found in specimens which are from a variety of geological environments and occur in serpentinites produced by different mechanisms, i.e. (1) crystallization in fractures to form vein serpentine, (2) pseudomorphic replacement of olivine and pyroxene to form mesh textures and bastites, and (3) recrystallization of pseudomorphic serpentine textures to produce non-pseudomorphic interlocking and interpenetrating textures. It is identified with the so-called "Povlen-type" chrysotile described in the literature.

Both polygonal and cylindrical fibres occur with unusually large diameters; probably the method of crushing and dispersion which was used hitherto to prepare samples for electron microscopy tended to break up the outer layers of fibres, whereas ion-thinning preserves the original features and dimensions.

ACKNOWLEDGEMENTS

The authors wish to thank Dr. P. E. Champness of this department, Dr. E. J. W. Whittaker, Department of Geology and Mineralogy, University of Oxford, and Dr. F. J. Wicks, Royal Ontario Museum, Toronto, for their comments on the manuscript.

The research was carried out during the tenure of a N.E.R.C. research studentship by B. A. Cressey. N.E.R.C. also supplied the electron microscope and ion-thinning machines.

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Manuscript received April 1976.